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ADDITIONAL PURIFICATION BY ULTRAVIOLET RADIATION OF RED SPD DYE AFTER ELECTROCHEMICAL DESTRUCTION

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ABSTRACT

Surface water pollution by industrial wastewater is one of the serious environmental problems of the Republic of Uzbekistan. A significant source of pollution is the textile industry, whose wastewater contains organic dyes and various related substances. This article presents the results of a study of the photooxidation of the active dye Red SPD after electrochemical degradation in an electrolytic cell with insoluble anodes. Studies have been carried out to determine the efficiency of active chlorine synthesis in the process of electrolysis of chloride solutions, the effect of active chlorine concentration, electrolyte composition, power consumption when using the combined electrochemical/ultraviolet irradiation process, and changes in the spectral characteristics of the dye solution for carrying out the photooxidation process have been studied. The technological parameters of the method of photooxidative post-treatment in treated water after electrochemistry are established, considering the concentration of oxidizing agents. The maximum current output of active chlorine occurs at a treatment time of 10 minutes. The use of NaCl as an electrolyte in comparison with Na_2SO_4 showed the best result in the purification process. When using NaCl with a concentration of 1.625 g L^{-1} in the composition of a sulfate-containing electrolyte, it can reduce the concentration of Red SPD to 1.0 mg L^{-1}. Photooxidative degradation can become an alternative for post-treatment of wastewater from textile enterprises. The developed technology confirms the efficiency of using the combined cleaning method.

Keywords: active dyes, active chlorine, electrochemical destruction, photooxidative post-treatment, wastewater.

INTRODUCTION

Textile production occupies a significant place in the industrial structure of the Republic of Uzbekistan. As is known, in the technological processes of textile factories, organic dyes are used, which are toxic substances and potential sources of pollution of natural waters. Active dyes are the most effective and large class of dyes that have many positive characteristics, such as brightness and purity of shades, resistance to wet treatments and light, due to which it is most often used in dyeing cotton, linen and viscose [1]. The production of fibre by the wet process is associated with the use of large amounts of water and the discharge of highly polluted wastewater, which is characterized by increased levels of colour, pH, turbidity, temperature, biochemical oxygen demand (BOD), chemical oxygen demand (COD) and toxic substances [2]. This type of wastewater can have a wide range of pH values, high concentrations of salts, namely Na_2SO_4 and NaCl, as well as carbonates [3].

Analysing the current state of the methods of wastewater treatment from dyes, it is necessary to note their diversity, but at the same time, the relevance of optimizing existing and searching for new highly efficient methods of wastewater treatment remains. The literature recommends the use of electrochemical processes for the removal of dyes from dyed textile effluents [4]. Electrochemical treatment is usually based on the removal of contaminants directly from the anode surface by the formation of $OH^{\cdot}$ and/or other oxidizing agents such as chlorine, persulfate, etc. However, the practice of operating wastewater treatment plants of textile enterprises using modern equipment and technologies shows that the concentration of pollutants entering the treatment plant increases by 30 - 40 % [5, 6]. This leads to a decrease in the performance of electrolysers (existing equipment), an increase in electricity consumption in the wastewater treatment process, and the need to install additional equipment. The presence of chloride ions in wastewater from textile industries intensifies the process of electrochemical degradation due to the formation of active chlorine, which has a large supply of chemical energy at the time of its formation. However, it has been found that the oxidizing capacity of the electrolysis products is not fully used in the purification process. Oxidative methods of wastewater treatment provide a more complete destruction of complex organic substances under the influence of oxidizing agents to simple compounds that are easily assimilated by microorganisms during biological treatment or in the processes of self-purification of water bodies [7 - 9]. The main feature of such methods is that they are based on two successive stages: the generation of active particles and their interaction with pollutants dissolved in water. Compared to traditional methods of water purification from organic compounds, such as combustion, oxidative methods in most cases consume less energy. Oxidative processes are characterized by a variety of options for the generation of hydroxyl radicals, which are extremely reactive species with rate constants of reactions with most organic molecules of the order of 10^6 - 10^9 $M^{-1}s^{-1}$ [10]. The generation of hydroxyl radicals is carried out in the process of various reactions, the conditions of which include a combination of chemicals (O_3, H_2O_2, transition metals, oxides) and auxiliary energy sources (UV radiation, electric current, ultrasound).

The use of post-treatment technology for wastewater from textile enterprises based on combined oxidative processes of electrochemical and photo-oxidative methods will generally reduce the specific energy consumption per unit of pollutants, achieve deeper destruction of difficult-to-oxidize organic pollutants in the presence of an excess concentration of active chlorine.

The purpose of this study is to study the method of photooxidative degradation to improve the efficiency of the facilities for electrochemical wastewater treatment of textile enterprises.

EXPERIMENTAL

For the experiment, a sample of Red SPD textile dye was taken, the concentration of the dye solution was determined on a UV- Vis spectrophotometer (Libra S35) by constructing a calibration curve at λ_{max} = 543 nm.

For the first stage of purification, a diaphragm electrolyser was used, representing a cylindrical body, where perforated anodes are made of titanium coated with ruthenium and titanium oxides (ORTA) and perforated cathodes made of unalloyed steel are coaxially located. The anode and cathode chambers are separated by an inert membrane, which provides a hermetic separation of the spaces. The anode chamber has a volume of 2.0 L, the cathode chamber - 1.0 L. The anode area is 335 cm^2.

For the second stage of the experiments, a laboratory non-flow photochemical reactor with an external radiation source was assembled. A low-pressure mercury lamp with a maximum emission mainly at 254 nm (55W, Philips TUV T8) was used as a light source. The radiation intensity of the UV lamp was 70 mW cm^{-2}. The UV lamp is installed parallel to the reflector in the upper part of the photoreactor at 200 mm from the working surface of the solution. An air-cooling system with electric fans is used to prevent overheating of the lamps and to remove heat outside the reactor. The inner surface of the photoreactor is made of polished stainless steel for maximum light reflection. The air-cooling system with electric fans does not maintain the temperature of the solution. The solution to be treated is placed in a Petri dish and located at a certain distance from the radiation source. During irradiation, the solution is stirred with a magnetic stirrer at a constant speed. If necessary, the heating function to a certain temperature is activated.

The processing of a model solution of the active dye Red SPD with a concentration of 100 mg L^{-1} was carried out in the anode chamber of the electrolyser, the concentration of NaCl/ Na_2SO_4 - 0.825-6.625 g L^{-1}, the concentration of sodium carbonate was 0.35 g L^{-1}, the initial pH = 10 -11. The current density is 200 A m^{-2}. UV treatment was carried out at 254 nm, 55W.

The bleaching efficiency was determined by the formula:

$$E = \left(\frac{C_0 - C_t}{C_0}\right) \times 100 \qquad (1)$$

where C_0 is the initial dye concentration, mg L^{-1}, C_t - dye concentration during treatment t, mg L^{-1}.

The measurements were carried out immediately after treatment at the wavelength of the visible maximum absorption of the dyes.

RESULTS AND DISCUSSION

Determination of technological parameters of the process of photooxidative post-treatment

The results of a study on the electrochemical degradation of the Red SPD dye are presented in our work [11]. It was previously established in this work that the destruction of the Red SPD dye whit a concentration of 100 mg L^{-1} becomes quite efficiently at a current density of 200 A m^{-2}, achieving bleaching to the standard indicators of wastewater in the city sewer. It was also found that COD was reduced by 80 - 85 %. The study of the influence of the pH value and temperature of the processed dye solution on the efficiency of the electrochemical treatment process showed that an increase in temperature from 20°C to 45°C rises the efficiency of decomposition of dyes and auxiliary organic substances, and the degree of oxidation of organic impurities in a slightly acidic medium is higher (pH = 4.8 - 5.0) than at pH 8.0 - 10.

The concentration of electrolytes (NaCl and Na_2SO_4) varied from 0.825 to 6.625 g L^{-1}, while the treatment process is most effective at a NaCl concentration of 3.3 g L^{-1}. It has been established that in the process of electrochemical degradation (ED), excess electrogenerated oxidants (EO) are formed, which require subsequent deactivation.

In this work, we determined the possibility of using an excess of EO in the process of photooxidation, which was carried out after electrochemical destruction. Reducing the processing time at the ED stage will reduce the concentration (EO), and the use of photooxidation will reduce the excessive concentration.

Determination of the efficiency of EO synthesis in the process of electrolysis of chloride solutions

The indicators are the current efficiency, % and the degree of conversion of chlorides into active chlorine (EO) NaCl, %. It is known that with an increase in the amount of electricity passed through a chloride-containing solution, the amount of electrogenerated active chlorine increases and, accordingly, the degree of decomposition of sodium chloride increases.

As can be seen from Fig. 1, the current efficiency of active chlorine has a pronounced maximum at a treatment time of 10 minutes. The low efficiency of active chlorine formation, observed in the first five minutes of electrolysis, is associated with the formation of hypochlorite in an alkaline medium and with the consumption of electricity for the side process of the formation of chlorates or perchlorates according to the Eq. 2 and Eq. 3 [12]:

$$6HClO + 3H_2O \rightarrow 2ClO_3^- + 4Cl^- + 12H^+ + 3/2O_2 + 6e^- \quad (2)$$

$$ClO_3^- + H_2O \rightarrow ClO_4^- + 2H^+ + 2e^- \quad (3)$$

A monotonous decrease in current efficiency with an increase in power consumption is associated with competing reactions of chlorine and oxygen evolution, as well as with depletion of the solution in Cl^- ions. Thus, it is advisable to carry out ED Red SPD with a processing time of 5 - 10 min.

To determine the possibility of using photooxidative degradation (UV) for additional purification of the dye solution after electrochemical treatment, experiments were carried out at different times of electrochemical degradation.

After 5 min of electrodestruction (ED_5), the first 5 min of UV treatment pass at a high speed, but then the

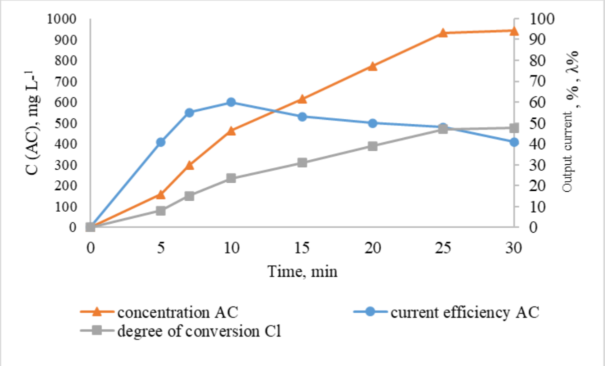

Fig. 1. Dependences of the concentration of electrogenerated active chlorine, current efficiency and the degree of conversion of chlorides, on the processing time in electrolyte whit composition: 3.3 g L^{-1} NaCl, 0.35 g L^{-1} Na_2CO_3, pH - 10.78.

treatment efficiency changes slightly, which is associated with an insufficient concentration of electrogenerated active chlorine (Fig. 2). The rate of discoloration during UV treatment increases significantly with increasing ED time from 5 to 7 min, and after ED_7 changes slightly (Fig. 2). Although the total processing time for $ED_7 + UV_{10}$ and $ED_{10} + UV_7$ is the same, it is obvious that it is economically feasible to carry out the first stage of treatment for 7 min and the second one for 10 min.

The total efficiency of ED + UV cleaning is higher with a shorter total processing time (Fig. 3).

Comparison of the use of residual active chlorine formed during ED for post-treatment of the electrotreated solution with UV treatment and without UV treatment showed the efficiency of the photooxidation process (Fig. 4).

Determination of the effect of active chlorine concentration on the efficiency of the photooxidation process of an electrotreated red SPD solution

Determination of the influence of the active chlorine concentration on the efficiency of the process of photooxidation of the electrotreated Red SPD solution was carried out using the anolyte as a source of active chlorine. Data on photooxidation are shown in Fig. 5.

With an active chlorine concentration of 16 mg L^{-1},

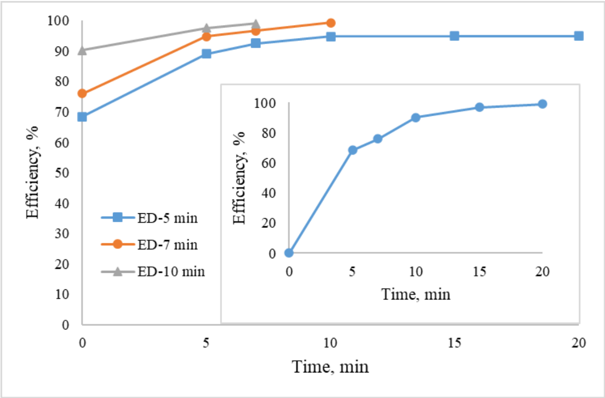

Fig. 2. Dependence of the bleaching efficiency of the electrotreated dye solution on the time of UV treatment. On the tab, the efficiency of decolorization of Red SPD during ED (3.3 g L^{-1} NaCl, 0.35 g L^{-1} Na_2CO_3, pH - 10.78).

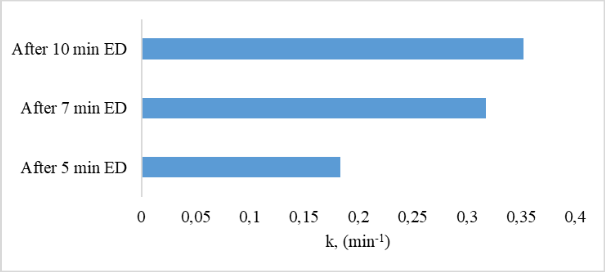

Fig. 3. Kinetic rate constants of UV destruction of the electrotreated dye solution at different ED times.

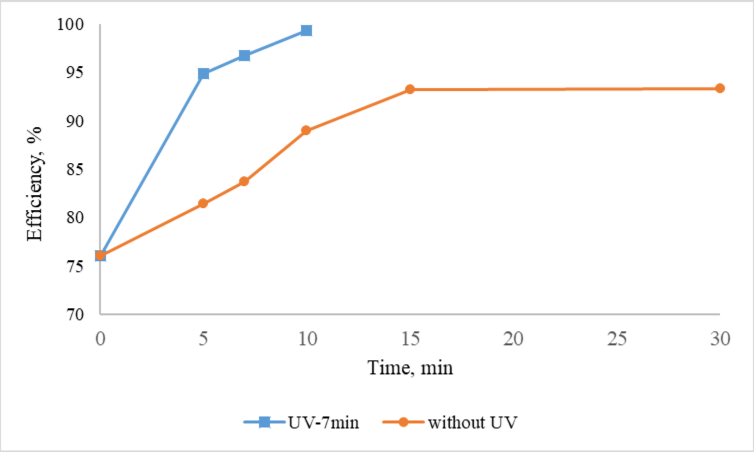

Fig. 4. Comparison of the post-treatment efficiency of the electrotreated Red SPD dye solution with UV treatment and without UV treatment.

even with a treatment time of 50 min, the treatment efficiency is 97.97 %, it can also be seen that in the last 10 min, the speed of the bleaching process decreases sharply. With an active chlorine concentration of 32 mg L^{-1} and a treatment time of 25 min, the bleaching efficiency is 98.6 %. The highest rate of oxidation, close to post-ED treatment, is observed at an active chlorine concentration of 64 mg L^{-1} (Fig. 5). High bleaching efficiency is achieved with a ratio of C_{dye}:C_{AC} = 1:2.9.

Influence of the composition of the electrolyte on the UV post-treatment process

When only sodium sulfate was used as an electrolyte in the dyeing process, electrochemical treatment showed low efficiency, subsequent UV treatment also showed low efficiency (Fig. 6).

Due to the low efficiency of the ED + UV process using only sodium sulfate as an electrolyte, it was necessary to determine the minimum concentration of introduced chlorides for effective processing (Fig. 7).

Sulfate ions have a significant impact on the effectiveness of ED. Sulfate ions, having the same relative mobility in solutions as chloride ions, can be simultaneously discharged at the anodes, increasing the total current consumption for the yield of atomic chlorine [13]. The ED kinetic rate constant depends not only on the content of Cl^- and SO_4^{2-} in solution, but also on their ratio (Fig. 8).

The kinetic rate constant of ED increases in

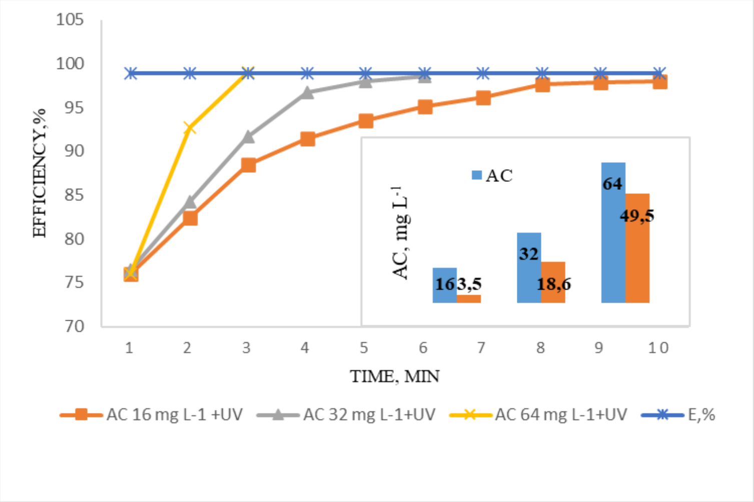

Fig. 5. Decolorization efficiency of Red SPD versus treatment time at various concentrations of electrogenerated active chlorine (AC). On the tab, the dependence of the residual concentration of AC on the initial concentration of AC after UV treatment (3.3 g L^{-1} NaCl, 0.35 g L^{-1} Na_2CO_3, pH - 10.78.), E - efficiency corresponding to the residual concentration of Red SPD < 1.0 mg L^{-1}.

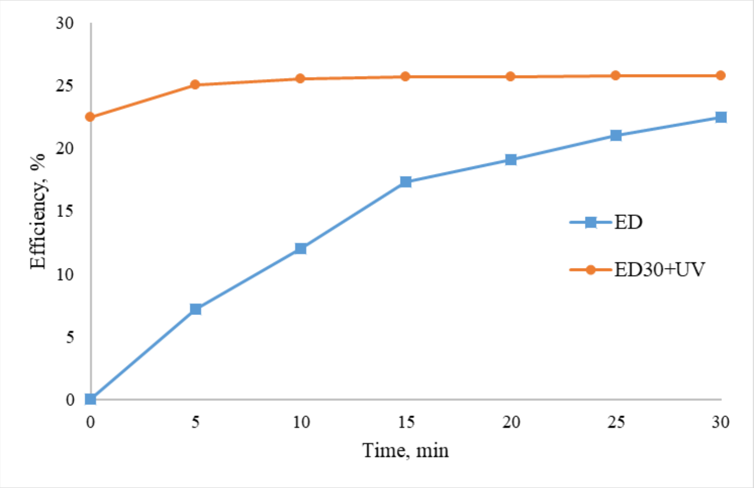

Fig. 6. Dependence of the dye bleaching efficiency during ED (stage 1) and UV treatment (stage 2) on the treatment time (3.3 g L^{-1} NaCl).

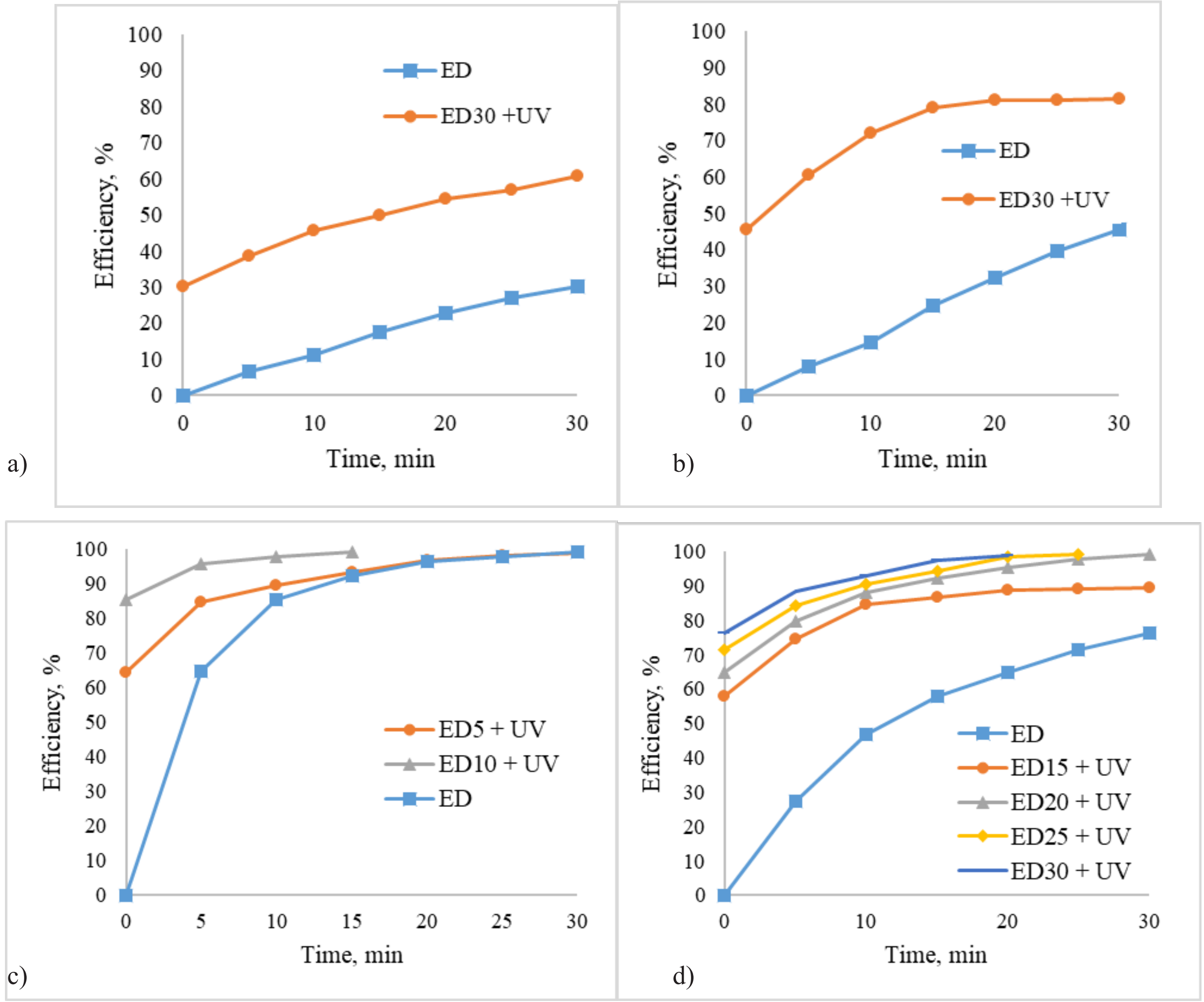

Fig. 7. Dependence of bleaching efficiency on ED+UV treatment time at different electrolyte concentrations (0.35 g L^{-1} Na_2CO_3, pH - 10.78). (a) NaCl - 0.413 g L^{-1}, Na_2SO_4 - 2.887 g L^{-1}, (b) NaCl - 0.825 g L^{-1}, Na_2SO_4 - 2.425 g L^{-1}, (c) NaCl - 1.625 g L^{-1}, Na_2SO_4 - 1.625 g L^{-1}, (d) NaCl - 2.425 g L^{-1}, Na_2SO_4 - 0.825 g L^{-1}

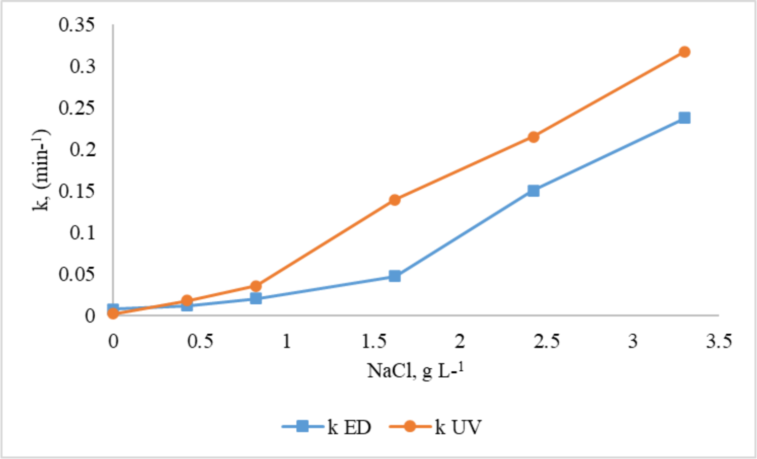

Fig. 8. Kinetic rate constants of ED and UV treatment with Red SPD at different concentrations of NaCl in the sulfate electrolyte.

proportion to the increase in NaCl concentration up to a concentration of 1.625 g L^{-1}. At the ratio Cl^- SO_4^{2-} in solution, the ED rate sharply increases. The rate of UV treatment increases significantly when NaCl concentration during ED is more than 0.825 g L^{-1}. However, the amount of electrogenerated active chlorine is not enough for complete bleaching with an ED efficiency of 45 % (Fig. 7).

In addition, the high colour of the processed solution is characterized by high optical density. In this case, the solution becomes less permeable to UV radiation. Therefore, $HClO^-$ can be irradiated with only a small portion of ultraviolet light to form fewer free radicals, and as a result, discoloration is reduced. Fig. 9 shows the optimum ED + UV settings to achieve maximum efficiency with minimum co-treatment time.

Thus, the concentration of NaCl - 1.625 g L^{-1} in the composition of the electrolyte containing sulfates allows, upon reaching the efficiency of the electrochemical instruction of 76 % and UV radiation within 20 min, to reduce the concentration of Red SPD in the treated water to 1.0 mg L^{-1} (Fig. 9).

Fig. 10 shows the change in the UV spectrum of the Red SPD dye solution during electrochemical degradation (stage 1) and UV treatment (stage 2).

The absorption in the visible region partially decreased during the ED time of 7 min and was almost completely removed after 10 min of UV treatment. The intensity of the peak at 295 nm continues to decrease, while at 265 nm and 400 nm it is absent. This may be due to the formation of active species such as OH • and Cl • according to the following scheme:

$$O_2 + e^- \rightarrow O_2^{\bullet -} \quad (4)$$

$$2O_2^{\bullet -} + H_2O \rightarrow 2OH^{\bullet} + OH^- + O_2 \quad (5)$$

$$Cl_2 + hv \rightarrow 2Cl^{\bullet} \quad (6)$$

$$Cl^{\bullet} + HR \rightarrow HCl + R^{\bullet} \quad (7)$$

It can be seen from reactions (4) - (7) that the increase in the bleaching efficiency during UV treatment can be explained by the formation of radicals from the electrogenerated $Cl_2^{\cdot}/HClO^{\cdot}$, which have a high oxidizing ability [14, 15]. With an increase in the time of UV treatment, a more complete destruction of the dye is achieved, however, long-term treatment is not economically feasible.

Change in COD value during ED+UV treatment

Even though the decolorization efficiency of the Red SPD dye solution is an important characteristic of the ED + UV process, it does not provide information about the decrease in the concentration of organic substances present in the solution, which is determined by the value of chemical oxygen demand (COD) [16]. Therefore,

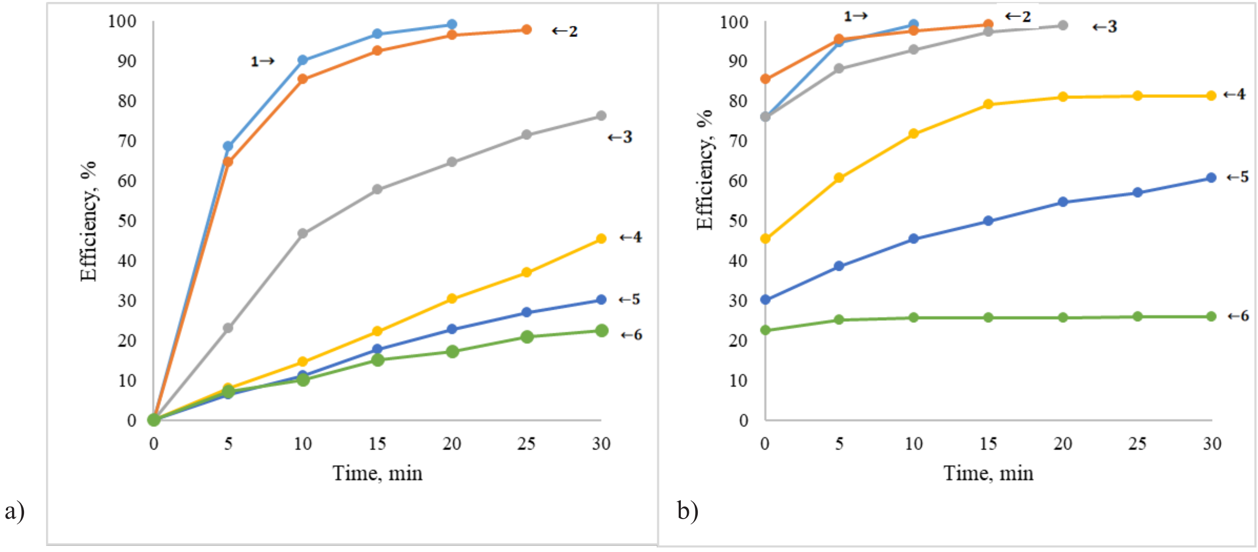

Fig. 9. Efficiency of ED (a) and UV treatment (b) at different concentrations of NaCl in an electrolyte containing sulfates (0.35 g L^{-1} Na_2CO_3, pH - 10.78). (a) 1) NaCl - 3.3 g L^{-1}; 2) NaCl - 2.425 g L^{-1}, Na_2SO_4 - 0.825 g L^{-1}; 3) NaCl - 1.625 g L^{-1}, Na_2SO_4 - 1.625 g L^{-1}; 4) NaCl - 0.825 g L^{-1}, Na_2SO_4 - 2.425 g L^{-1}; 5) NaCl - 0.413 g L^{-1}, Na_2SO_4 - 2.425 g L^{-1}; 6) Na_2SO_4 - 3.3 g L^{-1}, (b) 1) NaCl - 3.3 g L^{-1}, ED - 7 min; 2) NaCl - 2.425 g L^{-1}, Na_2SO_4 - 0.825 g L^{-1}, ED - 10 min; 3) NaCl - 1.625 g L^{-1}, Na_2SO_4 - 1.625 g L^{-1}, ED - 30 min; 4) NaCl - 0.825 g L^{-1}, Na_2SO_4 - 2.425 g L^{-1}, ED - 30 min; 5) NaCl - 0.413 g L^{-1}, Na_2SO_4 - 2.425 g L^{-1}, ED - 30 min; 6) Na_2SO_4 - 3.3 g L^{-1}, ED - 30 min

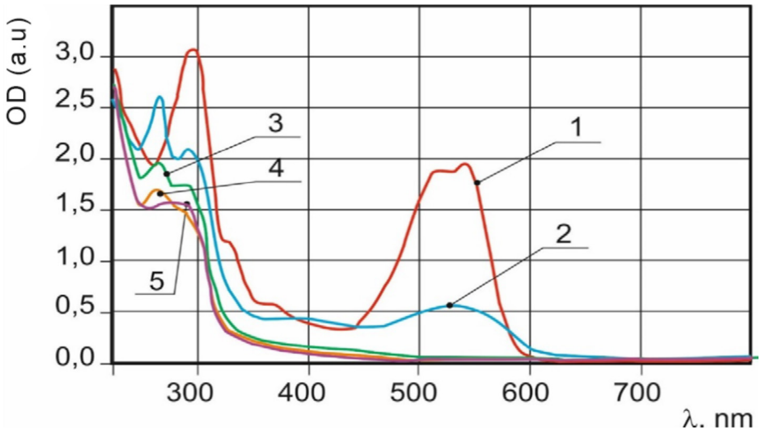

Fig. 10. Absorption spectra of the Red SPD dye at ED+UV (3.3 g L^{-1} NaCl, 0.35 g L^{-1} Na_2CO_3, pH - 10.78). 1 - Treatment time 0 min, 2 - ED - 7 min, 2 - UV- 5 min, 4 - UV - 10 min, 4 - UV- 15 min, 5 - UV - 20 min.

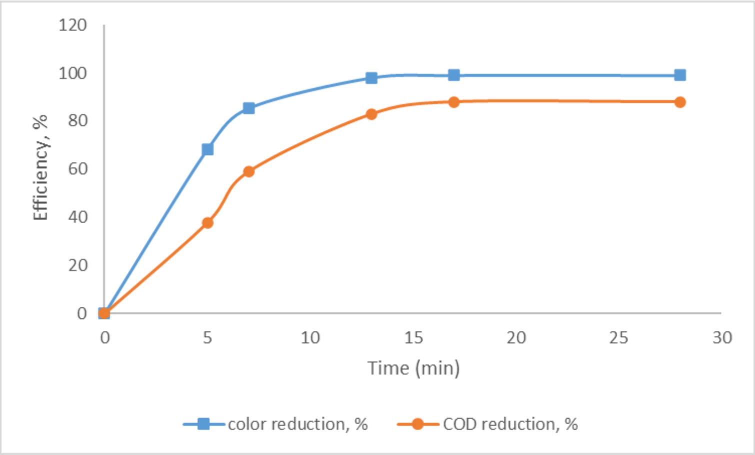

Fig. 11. Dependence of the effectiveness of color reduction and reduction of COD Red SPD on time in the process of ED + UV (3.3 g L^{-1} NaCl).

studies have been carried out to reduce the COD value during the combined cleaning process.

Comparison of bleaching efficiency and COD reduction suggests that Red SPD bleaching is more effective than COD reduction, this is due to the rather complex structure of the dye molecule, while the COD reduction efficiency is 88 %.

Energy consumption when using the combined ED + UV decolorization process Red SPD

Energy consumption is one of the important factors that determine whether the Red SPD combined ED + UV bleaching process can be used. The energy consumption for the combined process will be determined from the sum of the energy consumption for ED and UV treatment.

The specific energy consumption for ED is determined by the formula (8):

$$E_{ED} = \frac{U \times I \times t_e}{V} \quad (8)$$

where U - voltage, V, I - applied current, A, t_e - electrolysis time, h, V - the dye solution volume, L.

Electricity consumption during UV treatment [22] is calculated by Eq. (9):

$$E_{UV} = \frac{n\theta At}{V} \quad (9)$$

where n the number of lamps, Θ - radiation energy per unit area of the lamp, this information is provided by the equipment manufacturer, A - lamp area, t – time, V - the dye solution volume, L.

The energy consumption for decolorizing Red SPD during the ED and ED + UV process at various NaCl concentrations is shown in Fig. 11.

A reduction in energy consumption for the Red

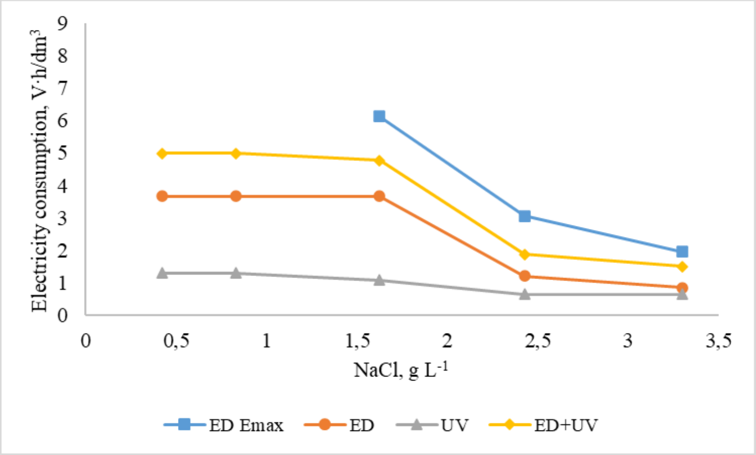

Fig. 12. Energy consumption for the destruction of Red SPD in the process of ED (ED E_{max}), in ED + UV by stages (ED, UV and ED + UV), (3.3 g L^{-1} NaCl, 0.35 g L^{-1} Na_2CO_3, pH - 10.78).

SPD combined bleaching process is observed at NaCl concentration $\geq$ 1.625 g L^{-1} (Fig. 11). The lowest value of power consumption is observed in the absence or a small number of sulfates in the composition of the electrolyte. Comparing the energy consumption for the ED process when a residual concentration of Red SPD < 1.0 mg L^{-1} (ED E_{max}) is reached and the total consumption for the ED + UV process, the energy consumption at a NaCl concentration $\geq$ 1.625 g L^{-1} is reduced by 22-28 %.

CONCLUSIONS

Thus, the technological parameters of the process of photooxidative post-treatment after electrochemical destruction were determined depending on the concentration of oxidizing agents in water containing dyes. It was determined that the maximum current efficiency of active chlorine has a pronounced maximum at a treatment time of 10 min. With an increase in power consumption, a monotonous decrease in current efficiency is observed, which is associated with competing reactions of chlorine and oxygen evolution, as well as with depletion of the solution in Cl^- ions.

Carrying out the electrodestruction process for 7 min only at a NaCl concentration of 3.3 g L^{-1} and subsequent UV treatment makes it possible to increase the bleaching efficiency due to the formation of radicals from the electrogenerated $Cl_2/HClO^-$, which have a high oxidizing ability. Using only sodium sulfate as an electrolyte in the dyeing process, electrochemical treatment showed low efficiency, subsequent UV treatment also showed low efficiency. It has been established that the concentration of NaCl - 1.625 g L^{-1} in the composition of the sulfate-containing electrolyte allows, with the efficiency of ED 76 % and UV for 20 minutes in the treated water, to reduce the concentration of Red SPD to 1.0 g L^{-1}.

Consequently, using ED at the first stage and UV treatment at the second stage, the specific energy consumption decreases with the high efficiency of Red SPD bleaching, which proves the effectiveness and economy of the combined cleaning method.

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